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(54) MIXED ESTERS OF 2,2-DIMETHYLPROPANE-1,3-DIOL AND LUBRICANT COMPOSITIONS

(71) We, RUHRCHEMIE AKTIENGESELLSCHAFT, a German Company, of Bruchstrasse 219, Oberhausen-Holten, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The invention relates to mixed esters of 2,2 - dimethylpropane - 1,3 - diol and lubricant compositions.

Lubricants for subsonic turbo-jet aircraft engines are required to possess the following essential properties:

Viscosities below 13 000 cSt at −54°C, viscosity indices above 138, flame points above 204°C and setting or pour points below −60°C (corresponding to USA Military Specification Mil—L—007808 F). Furthermore they must possess adequate thermal and oxygen stability.

Owing to their quaternary carbon atom, the esters of neopentyl glycols, as for example 2,2 - dimethylpropane - 1,3 - diol, 2,2 - diethylpropane - 1,3 - diol, 2 - methyl - 2-propyl - propane - 1,3 - diol, 2 - methyl - 2-butylpropane - 1,3 - diol, and aliphatic monocarboxylic acids exhibit favourable resistance to high temperature and oxidation, which qualify them for the application as lubricants, hydraulic oils and central hydraulic liquids at high temperatures. However, they do not meet the other requirements hereinbefore mentioned.

Neopentyl glycol esters derived from straight chain mono-carboxylic acids, for example pelargonic acid or capric acid, have viscosity indices within the required range but they have setting points of −35°C and −27°C respectively. The neopentyl glycol ester derived from the straight chain C_7-carboxylic acid has, indeed, a setting point of −62°C, but its viscosity index is insufficient.

Neopentyl glycol esters derived from branched chain monocarboxylic acids for example alpha-alkylhexanoic acids, 3,5,5 - trimethylhexanoic acid or isodecanoic acid respectively likewise do not meet the said requirements. Their setting points do fall within the specified range, but their viscosity indices are only in the range 80 to 90 and their viscosities at low temperatures are too high.

It is an object of the invention to provide carboxylic acid esters of a neopentyl glycol suitable as lubricants or as additives to lubricants which meet the hereinbefore mentioned requirements.

It has now been found that mixed esters obtained by the simultaneous esterification of 2,2 - dimethylpropane 1,3 - diol with a certain straight chain and a certain branched chain monocarboxylic acid possess the essential properties required for lubricants for turbo-jet aircraft engines with respect to viscosity-temperature behaviour, viscosity at low temperatures and setting point.

According to the invention, there is provided a diester of 2,2-dimethylpropane in which one of the acyl groups is that of a straight chain monocarboxylic acid and the other is that of a branched chain monocarboxylic acid, each acyl group containing from 4 to 12 carbon atoms. The invention also includes a mixture of two or more of the diesters.

The invention also comprises a lubricant based on carboxylic acid esters comprising at least one mixed ester of 2,2-dimethylpropane-1,3-diol and a straight chain monocarboxylic acid as well as a branched chain monocarboxylic acid having 4 to 12 carbon atoms in the molecule, alone or in admixture with other lubricants known in the art and with the conventional additives.

The preferred esters are those of straight chain and branched chain monocarboxylic acid having from 5 to 10 carbon atoms in their molecules.

An outstanding combination of desired properties are shown by the esters, 2,2-dimethylpropane - 1,3 - diol - 3,5,5 - trimethylhexan-

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ate - pelargonate and 2,2 - dimethylpropane-1,3 - diol - 2¹ - ethylhexanate - pelargonate. They possess low viscosities at −54°C, viscosity indices of above 140, setting points of about −70°C and flame points above 204°C.

The esters according to the invention can be prepared in conventional manner.

The esters according to the invention may be admixed with other liquid materials, for example, conventional fluid bodies suitable for use as lubricants, in any desired ratio, preferably one or more ester lubricants, for example esters of dihydric alcohols and monocarboxylic acids as well as esters of dicarboxylic acids and monohydric alcohols known in the art. Furthermore they may be admixed with mineral oil lubricants as well as lubricants which are organosilicon compounds, polyphenylether oils and phosphoric acid esters.

The properties of two mixed esters of 2,2-dimethylpropane - 1,3 - diol with straight chain and branched chain monocarboxylic acids according to the invention are shown in Table A. The properties of uniform diesters of 2,2-dimethylpropane - 1,3 - diol with straight chain monocarboxylic acids on the one hand and with branched chain monocarboxylic acids on the other hand as well as the properties of bis - (2 - ethylhexyl) sebacate, known in the art as a lubricant for turbo-jet aircraft engines, have also been set out in Table A by way of comparison.

TABLE A

	Ester	Viscosity in cSt			Viscosity index	Setting point °C.	Flame point °C.
		37.8°C.	−40°C	−54°C			
Ester with straight chain monocarboxylic acids	2,2-Dimethylpropane -1,3-diol-di-oenanthic acid ester	5.9	410	—	116	−62	204
	-di-pelargonic acid ester	9.2	solid	—	140	−35	220
	-di-capric acid ester	11.3	solid	—	145	−27	235
with branched chain monocarboxylic acids	2,2-Dimethylpropane -1,3-diol--di-3,5,5-trimethyl hexanate	14.6	8410	214530	88	−60	200
	-di-ethylhexanate	8.51	1815	15125	88	−69	197
Esters with straight chain as well as branched chain monocarboxylic acids	2,2-Dimethylpropane -1,3-diol-3,5,5-trimethyl-hexanate-pelargonate	10.8	1372	10400	146	−70	210
	2,2-Dimethylpropane-1,3-diol-ethylhexanate pelargonate	9.3	867	9100	142	−70	205
Reference ester as representative lubricant for turbo-jet aircraft engines	bis-(2-Ethylhexyl) sebacate	12.5	1300	10200	152	−70	232

The invention is illustrated in the following example.

Example

A round-bottom flask of 10 litre capacity was charged with 1352 grams 2,2 - dimethylpropane - 1,3 - diol, 2157 grams perlargonic acid and 2157 grams isononanoic acid (3,5,5-trimethylhexanoic acid). To the mixture were added 500 cc cumene as entrainer and 1 ml concentrated sulphuric acid as catalyst. The solution was heated under a reflux condenser for 5 hours with stirring, the temperature being held at 153°C. During this time the theoretical amount of reaction water of 468 ml was removed by the entrainer and separated with the aid of a laterally provided water separator. Cumene was separated from the reaction product by distillation under the vacuum of a water-jet pump. Unreacted and partially reacted components were separated from the cumene-free reaction product in two fractions by vacuum distillation. A first fraction of 315 grams comprising the acid-containing components distilled over between 55°C and 127°C at 0.02 torr. A second fraction of 370 grams was obtained between 110°C and 150°C at 0.003 torr. The distillation residue of 4500 grams was the desired raw ester oil. Thus the yield amounted to 86.6%. The neutralisation number of the ester was 0.1 mg KOH per gram, and its ester number was 292 mg KOH per gram. The raw product was distilled at 0.001 torr down to a residue of 150 grams, the mixed ester 2,2 - dimethylpropane - 1,3-diol - 3,5,5 - trimethylhexanate - pelargonate being obtained as a water-white product. It gave the following characteristic data.

Density d_4^{20} =	0.916
Viscosity in cSt	
at 98.9°C	2.94
37.8°C	10.7
−40°C	1410
−54°C	10300
Viscosity index	146
Setting point °C	−70
Flame point °C	210

WHAT WE CLAIM IS:—

1. A diester of 2,2 - dimethylpropane - 1,3-diol in which one of the two acyl groups is that of a straight chain monocarboxylic acid and the other is that of a branched chain monocarboxylic acid, each acyl group containing from 4 to 12 carbon atoms.

2. A diester according to claim 1, in which each acyl group contains from 5 to 10 carbon atoms.

3. 2,2 - Dimethylpropane - 1,3 - diol 3,5,5-trimethylhexanate - pelargonate.

4. 2,2 - Dimethylpropane - 1,3 - diol 2'-ethylhexanate-pelargonate.

5. A mixture comprising two or more of the diesters claimed in any one of the preceding claims.

6. A lubricant based on carboxylic acid esters comprising at least one mixed ester of 2,2 - dimethylpropane - 1,3 - diol and a straight chain monocarboxylic acid as well as a branched chain monocarboxylic acid having 4 to 12 carbon atoms in the molecule, alone or in admixture with another lubricant known in the art and with the conventional additives.

7. A lubricant comprising at least one of the diesters claimed in any one of claims 1 to 4 and another lubricant.

8. A lubricant comprising at least one of the diesters claimed in any one of claims 1 to 4 and a lubricant additive.

9. A lubricant according to claim 8, including another lubricant.

10. A lubricant according to claim 7 or claim 9, in which the other lubricant is an ester lubricant.

11. A lubricant according to claim 10, in which said ester lubricant is an ester of a dihydric alcohol with a monocarboxylic acid or an ester of a monohydric alcohol with a dicarboxylic acid.

12. A lubricant according to claim 7 or claim 9, in which the other lubricant is a mineral oil, an organosilicon compound, a polyphenyl ether oil or a phosphoric acid ester.

13. A lubricant or hydraulic fluid comprising a diester according to claim 1, substantially as hereinbefore described.

EDWARD EVANS & CO.,
53—64 Chancery Lane,
London, W.C.2.
Agents for the Applicants.

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